

Environmental Technologies Group ROCKY FLATS SOLARPOND/PONDCRETE PROJECT

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November 6, 1992



Mr. Edward M. Lee, Jr. Program Manager Solar Ponds Remediation Program EG&G Rocky Flats, Inc. Building 080 P. O. Box 464 Golden, Colorado 80402-0464

Subject: Rocky Flats Plant Solar Evaporation Ponds Stabilization Project [WBS 710 PROJECT MANAGEMENT - HALLIBURTON NUS ROCKY FLATS]

COMMENTS TO DOE EVALUATION OF WASTE CHARACTERIZATION REPORT & DESIGN

CRITERIA - RF-HED-92-0771

Dear Mr. Lee:

We received a letter from F. Lockhart dated October 23, 1992 yesterday requiring responses to DOE comments on the referenced deliverable documents. These are enclosed for your use in responding to the DOE RFQ.

One of the problems appears that the DOE is currently reviewing initial/draft copies of documents. In some cases the final document was transmitted to EG&G three months ago. It may prove beneficial to coordinate with review teams to ensure that current documents are being used. We can provide EG&G a current status of all deliverable documents if required.

I hope these responses appear complete. With only one day to respond, we have quickly provided our comments to the DOE audit.

If we can provide any additional responses, please advise.

Sincerely,

HALLIBURTON NUS ENVIRONMENTAL CORPORATION

Ted A. Bittner Project Manager

TAB/jg

Enclosure

cc: S. Heiman

A:\LTR\LEE34 RF-HED-92-0771

A-0U04-000423

ADMIN RECORD

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REVIEWED FOR CLASSIFICATION

Date 🚣



### INTERNAL CORRESPONDENCE

C-49-11-2-054

MEMO TO: TED BITTNER

DATE: NOVEMBER 6, 1992

FROM:

RICH NINESTEEL

cc: M. SPERANZA

SUBJECT:

ROCKY FLATS SOLAR POND PROJECT

T. SNARE

RESPONSE TO DOE COMMENTS ON

R. SIMCIK

PONDCRETE WASTE CHARACTERIZATION REPORT

J. SCHMIDT

J. D. CHIOU

ROCKY FLATS PROJECT NO. 2K68.224A

FILE: 2K68

Attached please find the preliminary responses to DOE's comments concerning the Pondcrete Waste Characterization Report (memorandum from A. Rampertraap to F. Lockhart dated September 30, 1992). The responses were propared in one day to help meet DOE's requested turnaround time. As such, please consider the responses preliminary until a final review can be done next week.

### Please note the following:

- The Internal Draft report was reviewed. We have subsequently issued Revision 0, which contains some revised text. I suggest a copy be forwarded to the reviewer immediately.
- The reviewers main concern was with the sampling rationale. Since this was explained in much more detail in the Sampling and Analysis Plan for Pondcrete and Saltcrete, I suggest a copy be forwarded to the reviewer immediately. Pertinent sections are attached.
- If the responses to comments are still not sufficient to meet the needs of the reviewer, I suggest a meeting or a conference call be arranged with our staff (in particular Dr. Chiou) to discuss the matter further.

Please call me if there are any further questions.

RN/pam

Attachments

By C. 111. Pasaua UNU

Date 11 - 5 - 92

## PRELIMINARY (11/6/92) RESPONSE TO COMMENTS BY DOE EM-453 ON THE PONDCRETE WASTE CHARACTERIZATION REPORT

### CRITICAL COMMENT

The following responses should clarify the logic used to select the number of pondcrete samples needed to characterize the population of billets:

- The sampling rationale presented in the Pondcrete Waste Characterization Report was a condensed version of the rationale presented in the Sampling and Analysis Plan for Pondcrete and Saltcrete, Combined Deliverable Number 211B, 211C, 221B, and 221C (HALLIBURTON NUS, October 1991). The reviewer is referred to the pertinent sections of this document for a better understanding of the sampling rationale (see attachment).
- As stated in the Sampling and Analysis Plan, previous ponderete data was statistically analyzed to determine the relative standard deviations (RSDs) and the possible worst case concentrations (97.5% upper tolerance limits) of selected parameters. The RSD is a measurement of the variability of a parameter in a population. The sample size required to characterize a population to a specified confidence level is related to the variability of the population. For example, a population with higher variability would require more samples to characterize it than a population with lower variability for the same level of confidence.

The main objective of the waste characterization program was to support the development of a successful stabilization recipe, not to characterize every single chemical constituent to the same level of confidence regardless of its importance to the project.

The data showed that some parameters had relatively high RSDs. However, not all parameters are of equal importance as they relate to the development of stabilization recipes. More specifically, many parameters that had high RSDs were not present at concentrations that were of concern, either from a regulatory standpoint or a chemical engineering standpoint for cement stabilization. Therefore, it was not a sound engineering approach to base the sampling program on parameters that had little relevance to the problem. Instead, it was determined that the most likely mode of failure of the stabilization mixes would involve the leaching of hazardous constituents, using the TCLP test, above regulatory standards, in this case the applicable Land Disposal Restrictions (LDRs). The leachate concentrations were conservatively estimated for key parameters and then compared to the parameter's LDR standard. This comparison allowed an evaluation of which parameters had the highest probability of exceeding their respective standards. These parameters would therefore be the most important for the development of a successful stabilization recipe.

For both triwalls and metal containers, cadmium had the highest leachate/standard ratio, by at least an order of magnitude over the next contaminant. Cadmium was thus chosen as the parameter whose statistical data would be used to determine the number of samples to characterize the populations of ponderete. All other contaminants were not present at concentrations that were of regulatory or engineering concern. If a contaminant of lesser

concern but higher RSD was used to estimate the number of samples for ponderete characterization, excess samples would have been required, with no benefit to the project.

### SPECIFIC COMMENTS

- 1. The first goal stated in Section 1.3 was to "characterize the two population forms of pondcrete."

  This statement can be expanded to include an assessment of regulatory compliance, including the LDR standards applicable to the waste.
- 2,3. Please see the response to the CRITICAL COMMENT and the section of the Sampling and Analysis Plan appended to this memorandum.
- 4. HALLIBURTON NUS was not provided with production data, therefore it is not possible to accurately determine whether the production dates of the sampled billets are proportional to the rate of production. This problem is further exacerbated by the lack of accumulation date data for some of the billets.
- 5. This comment is acknowledged. However, the two sections were presented in the order in which they appear because the laboratory analysis (Section 2.3.1) precedes data validation (Section 2.4). As a compromise, we propose adding references to Section 2.4 in Section 2.3.1 where definitions are required.
- 6. The commenter reviewed the Internal Draft of the subject report. The sentence referenced in this comment no longer appears in Revision 0, which was issued in September 1992. Section 4.2 in Revision 0 discusses the salt content of the two ponderete populations.
- 7. The referenced statement from the Internal Draft report no longer appears in Revision 0. The average data do not show a significant difference between the two populations of pondcrete for moisture (gravimetric) or cement constituents (calcium, iron, aluminum). However, the chemical data are not capable assessing the efficiency of the mixing and cement hydration, both of which are key to producing a stable waste form. It should also be noted that both populations of pondcrete were apparently produced with a high water/cement ratio, indicating that all pondcrete was probably deficient in cement content.
- 8. Section 4.2 has been modified in Revision 0. The data indicate that the two populations are similar based on comparison of average data for key parameters. Since the regulatory concerns were already discussed in Section 4.1, and considering the similarities presented in Section 4.2, the statement concerning methanol is reasonable.
- 9. The triwall sample that exceeded the LDR standard for amenable cyanide was PC-11500-T-D, which was produced on April 7, 1988. It should be noted that the duplicate of this sample did not exceed the LDR standard. The nearest triwall sample was PC-12503-T, which was produced April 18, 1988. This sample, as with all the other triwall samples, did not exceed the LDR standard for amenable cyanide. The method specified by SW846 shows that the triwall population as a whole does not exceed the LDR standard, and that no further sampling is required.

HALLIBURTON NUS

TEL:4129214040

Nov 06,92

12:57 No.005 P.05

ATTACHMENT

### 1.0 PROJECT DESCRIPTION

This plan describes the requirements for sampling several waste forms located at the Rocky Flats Plant in support of the Solar Pond/Pondcrete Stabilization project being conducted by HALLIBURTON NUS Environmental Corporation (HALLIBURTON NUS). The waste forms of concern are as follows:

- Solar pond sludge and water
- Pondcrete tri-walls

6 1 10

- Saltcrete tri-walls
- Clarifier sludge and water
- Evaporator bottoms (not currently available)

This Sampling and Analysis Plan will only address the Pondcrete and Saltcrete. The remaining waste sources are addressed in the Waste Sampling and Analysis Plan for Pond Sludge and Water, and Clarifier Sludge and Water (HALLIBURTON NUS, 1991).

The following sections contain descriptions of the waste forms to be sampled, the scope of sampling activities, and sampling strategy and rationale.

### 1.1 Site Description

### 1.1.1 Pondcrete

Department of Energy (DOE)/Rockwell began phasing out use of the solar evaporation ponds in the early 1980's because of environmental concern. The plan for cleanup of the ponds was to drain and treat the liquid waste and to mix the pond sediments/sludges with cement. The resulting solidified material known as Pondcrete was to be disposed of at DOE's Nevada Test site (NTS).

DELIVERABLE 211B, 211C, 221B, 221C POMDCRETE SAMPLING & AVALYSIS PLAN SALTCRETE SAMPLING & AVALYSIS PLAN REVISION 0 October 24, 1991

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Clean out of the largest surface impoundment (Pond 207A) began in 1985 with a pugmill process. The sludge from the bottom of the pond was pumped to a clarifier where it was allowed to settle out before being pumped to the pugmill. Cement was added to the sludge and mixed to a desired consistency by paddles attached to the auger shaft. The Pondcrete mixture was then fed through a chute into lined tri-walls. Improper mixing of cement and sludge resulted in some Pondcrete blocks that did not solidify properly or crumbled and cracked during storage pursuant to disposal at NTS.

Since the discovery of the Pondcrete problems in May 1988, DOE has not cleaned up any additional sediment from the solar ponds. Approximately 2,000 Pondcrete blocks had already been buried at NTS prior to the discovery of the problems. Since that time, 8,666 blocks have been inspected, approved, repackaged, and shipped to the test site for storage; and 8,031 blocks are awaiting remixing and repackaging. Substantial additional work remains to be done to clean up the ponds.

The Pondcrete blocks awaiting reprocessing are currently being stored in tension membrane structures (tents) on the 750 and 904 pad areas. Approximately 2550 of the Pondcrete blocks failed to solidify properly and the tri-wall containers are being stored in metal containers.

Pondcrete is a mixture of cement and sludge material generated from evaporating wastewater and is very high in salts, primarily calcium and potassium salts, with some sodium salts. Pondcrete has been sampled and analyzed several times for numerous compounds and parameters. The following provides a brief description of the chemical characterization of Pondcrete (Rockwell International, 1989).

1 6 5 10 1

### Volatiles

Only five volatile compounds registered above detection limits (ADL) in any of the Pondcrete samples analyzed. Information on those analytes are summarized as follows:

Volatile Analyte	Number of ADL Readings	Average of ADL Readings	Range of ADL Readings
Methylene Chloride	3 • of 30	16.9 ppb	7.3 to 35 ppb
Acetone	20 of 30	39.7 ppb	11 to 180 ppb
2-Butanone	9 of 30	16.7 ppb	12 to 23 ppb
Tetrachloroethene	10 of 30	20.2 ppb	5 to 73 ppb
1,1,2,2-Tetrachloroethane	1 of 30	160.0 ppb	

A series of three other samples indicated very high methylene chloride concentrations but were not included in the ADL readings shown because of very high concentrations in the blank also.

### Semivolatiles

Only four semivolatile compounds registered above detection limits in any of the Pondcrete samples analyzed. Information on those analytes is summarized as follows:

Semivolatile Analyte	Number of ADL Readings	Average of ADL Readings (ppb)	Range of ADL Readings (ppb)
2-Nitroaniline	1 of 30	970	
Di-n-Butyl phthalate	1 of 30	590	
Fluoranthene	8 of 30	722	374-1,683
Bis(2-ethylhexyl)phthalate	12 of 30	4,497	152-14,949

### Pesticides/PCBs

Of the two samples tested, all concentrations were below detection limits for the pesticide/PCB analytes.

### # Metals

Total metal analysis was performed on six Pondcrete samples. The results are summarized as follows:

Metals	Average Concentration (ppm)	Range of Concentrations (ppm)
Aluminum	27,330	16,820-33,400
Arsenic	8.98	4.11-24.6
Barium	600	205-2,000
Beryllium	54	1.16-77.6
Cadmium	390	8.16-590
Calcium	371,280	243,300-577,180
Chromium	278	176-420
Cobalt	30.9	20.9-33.8
Copper	155	23.4-236
Iron	13,620	9,730-17,620
Lead	29.6	2.38-43.0
Hagnesium	5,670	1,210-7,680
Manganese	2,090	804-6,910
Hercury	1.43	<0.02-2.32
Nickel	. 116	57.4-156
Potassium	157,840	9,470-329,300
Silver	13.4	6.63-23.4
Sodium	26,090	1,580-53,230
Vanadium	43.6	28.8-62.7
Zinc	113	62.1-210
Percent Solids	67.8%	44.48-948

### Cyanides

Analyses for total cyanide and cyanide amenable to chlorination were performed on samples taken from five different blocks of Pondcrete. A duplicate sample was taken from one of the blocks, therefore a total of six samples were analyzed. The results are summarized as follows:

DELIVERABLE 2118, 211C, 2218, 221C POMOCRETE SAMPLING & ANALYSIS PLAN SALTCRETE SAMPLING & ANALYSIS PLAN

Analysis	Average Concentration (ppm)	Range of Concentrations (ppm)
Total Cyanide	9.65	7.14-12.1
Amenable Cyanide	7.41	4.05-9.90

### Radiochemistry

Radiochemistry analyses were performed on five Pondcrete samples. The results are summarized as follows:

Analysis	Average Concentration (pCi/g)	Range of Concentrations *** (pCi/g)
Gross Alpha	2,400	1,700-3,800
Cross Beta	38	12-53
Pu-239	750	130-1,800
Am-241	1,000	690-1,600
U-233, U-234	44	33-60
U-238	48	40-66
Tritium	1.7 pCi/mL	1.5-2.1 pCi/ml

### Toxic Characteristic Leaching Procedure (TCLP)

• TCLP Volatiles. Three Pondcrete samples were subjected to the TCLP and analyzed for 21 volatile compounds. These are the volatile compounds that appear in 40 CFR 268.41, Table CCWE (Constituent Concentrations Waste Extract), for F001 through F005 spent solvents. Only three constituents were observed at concentrations above the detection levels and in each case, this occurred in only one out of three results. The three compounds and their single concentration above detection levels are as follows:

Compound	TCLP Concentration (ppb)
1,1,1-Trichloroethane	8
Tetrachloroethane	5
Toluene	60

It should be noted that toluene was also detected in the blank at 23 ppb.

• TCLP Semivolatiles. The same three Pondcrete samples were analyzed for the semivolatile compounds that also appear on the Table CCWE for F001, F002, F003, and F005 spent solvents. None of the four compounds considered (cyclohexane, 1,2-dichlorobenzene, pyridene, and 2-nitropropane) were observed at concentrations above detection.

In addition to the TCLP, the Pondcrete was also tested for ignitability, corrosivity, reactivity, and EP toxic metals. Pondcrete did not test positive for ignitability, corrosivity, or reactivity. Only one EP toxic metal tested positive for the Pondcrete samples. The Pondcrete was found to be toxic for cadmium in eight of the 26 samples. In four of the eight readings, the average was 16.4 mg/l, with a range of 1.5 mg/l to 42 mg/l. The EP toxicity standard for cadmium is 1 mg/l.

### Applicable EPA Hazardous Waste Numbers

The Pondcrete waste has its origin in a collection of wastewaters coming from approximately 30 different buildings, most of those....

DELIYERABLE 2118, 211C, 2218, 221C POMDORETE SAMPLING & AVALYSIS PLAN SALTCRETE SAMPLING & AVALYSIS PLAN with multiple contributing streams. The applicable EPA hazardous waste numbers for Pondcrete are as follows:

Hazardous Waste Number	Description	
D006	Toxic for cadmium	
F001	Spent halogenated solvents used in degreasing	
F002	Spent halogenated solvents	
F003	Spent nonhalogenated solvents	
F005	Spent nonhalogenated solvents	
F006	Wastewater treatment sludges from electroplating operations	
F007	Spent cyanide plating bath solutions from electroplating	
F009	Spent stripping and cleaning bath solutions from electroplating operations where cyanides are used	

### 1.1.2 Saltcrete

Saltcrete is generated by solidifying the nitrate salt residue from an evaporation process at the Liquid Waste Treatment Facility in Building 374. In very simplified terms, the 374 wastewater treatment operation can be broken into three processes. Depending on its radiological contamination and point of origin, wastewater can go straight into any one of the three treatment process; however, inside the facility, the processes are interrelated. The three basic processes are:

- (1) Evaporation
- (2) Flocculation/precipitation
- (3) Sludge dewatering

The flocculation/precipitation activity is designed for the removal of radioactive material. The settled sludge from this process goes to the sludge handling step and the overflow goes to the evaporator. The evaporator receives less contaminated wastewater directly. The residue or concentrated salt solution from the

evaporator is mixed with cement to immobilize particulates and remove the oxidizer and corrosive characteristics of the salt and/or concentrated salt solutions. The resulting waste form is referred to as Saltcrete (Rockwell International, 1989).

The wastewater now going to Building 374 includes that which previously went to the evaporation ponds from which Pondcrete was generated. Therefore, in general terms, the waste streams contributing to the formation of Saltcrete are similar to those identified for Pondcrete. Multiple sources/activities are involved (about 30 different buildings), generating wastewater with both radiological and hazardous chemical contaminants. The major distribution of wastewaters have radiological contamination below a specified level and are sent directly to the evaporator. Some of the processes generating wastewaters that are of particular concern from a RCRA standpoint include:

- (1) Various laboratory activities
- (2) Electroplating operations which include the use of cyanides
- (3) Metal machining/manufacturing including cleaning/ degreasing with solvents
- (4) Acid and caustic cleaning/rinsing solutions

The analytical results from various sampling events are described in the following paragraphs.

### Volatiles

Only six volatile compounds registered above detection limits in any of the 18 Saltcrete samples analyzed. Information on those analytes is summarized as follows:

Analyte	Number of ADL Readings	Average of ADL Readings	Range of ADL Readings
Acetone	15 of 15'	168 ppb	89-380 ppb
2-Butanone	15 of 15'	39 ppb	21-70 ppb
Benzene	1 of 15'	26 ppb	
Methylene chloride?	2 of 18	14 ppb	7.7-20 ppb
Tetrachloroethene	2 of 18	7 ppb	6-8 ppb
Toluene	15 of 15'	22 ppb	5.1-51 ppb

The Appendix III volatile analyses of samples taken August 1988 did not include these compounds; hence only 15 readings.

### <u>Semivolatiles</u>

Only three semivolatile compounds were detected above detection limits in any of the 18 Saltcrete samples analyzed. It should be noted that the semivolatile analyses of samples taken in August 1988 did not include any of the analytes observed ADL; therefore, the total number of readings is only shown as 15. Information on those analytes is summarized as follows:

Analyte	Number of ADL Readings	Average of ADL Readings	Range of ADL Readings
4-Chloro-3-methyl phenol	1 of 15	660 ppb	
Butyl benzyl phthalate	1 of 15	3,530 ppb	
Bis(2-ethylhexyl)phthalate	1 of 15	4,156	

The volatile analyses of samples taken in August 1988 were all positive for this analyte, but because method and extract blanks were also positive at similar values, these values were not included as ADL readings.

### <sup>∑</sup>Metals

Total metal analyses were performed on only one Saltcrete sample. The results are as follows:

Metal	Concentration (ppm)	
Aluminum	11,520	
Antimony	<6.96	
Armenic	4.04	
Barium	160	
Beryllium	0.70	
Cadmium	4.30	
Calcium	182,390	
Chromium	117	
Cobalt	19.8	
Copper	17.9	
Iron	14,290	
Lead	3.55	
Magnesium	2,860	
Manganese	606	
Mercury	<0.02	
Nickel	<0.02	
Potassium	30.4	
Selenium	<0.58	
Silver	8.94	
Sodium	4,870	
Thallium	<1.16	
Vanadium	38.3	
Zinc	61.5	

### Cyanide

Analyses for total cyanide and cyanide amenable to chlorination were performed on samples taken from four different blocks of Saltcrete. A duplicate sample was taken from one of the blocks; therefore, a total of five samples were analyzed. The results are summarized as follows:

DELIVERABLE 211B, 211C, 221B, 221C PONDORETE SUMPLING & ANALYSIS PLAN SALTORETE SAMPLING & ANALYSIS PLAN

Analysis	Average Concentration (ppm)	Range of Concentrations (ppm)
Total Cyanide	15.5	12.6-18.5
Amenable Cyanide	13.2	6.2-18.2

### Radiochemistry

Radiochemistry analyses were also performed on only a single Saltcrete sample. The results are as follows:

Analysis	Concentration (pCi/g)*	
Gross Alpha	240 +/- 60	
Cross Beta	170 +/- 60	
Pu-239	160 +/- 10	
Am-241	88 +/- 4	
U-233, U-234	25 +/- 10	
U-238	88 +/- 18	
Tritium	1.3 +/- 0.3 (pCi/mL)	

<sup>\*</sup>Plus or minus (+/-) values indicate the 95 percent confidence range for the reported values.

### RCRA Characteristics

TCLP analysis was conducted on three Saltcrete samples for volatiles, acids, and methanol (i.e., the compounds associated with F001 through F005 wastes). Acetone and methylene chloride were detected in the low ppb range (10 to 25 ppb) for the extract, however these compounds were also in the extract blank. Methyl isobutyl ketone, 2-butanone, and toluene had estimated readings below the detection limit of 10, 10, and 5 ppb, respectively.

EP Toxic Metals analysis were taken on 13 samples in April 1988.

All analytical results for EP Toxic Metals except lead were below the following detection limits:

Hetal	Detection Limit (ppm)	EP Toxicity Limit
Arsenic	0.10	5.0
Barium	1.0	100.0
Cadmium	0.05	1.0
Chromium	0.5	5.0
Lead	0.5	5.0
Mercury	0.005	0.2
Selenium	0.1	1.0
Silver	5.0	5.0

Lead was observed in a single sample at a concentration at the detection limit (0.5 ppm). Two samples taken within one month of each other in 1986 provided variable information. The first provided positive readings for five metals while the second had less than detectable for all eight metals. These results are summarized as follows:

	Concentr	ation (ppm)
Metal	lst 1986 Sample	2nd 1986 Sample
Barium	0.30	<20.0
Cadmium	0.092	<0.2
Chromium	2.99	<1.0
Lead	0.33	<1.0
Silver	0.050	<1.0

Saltcrete was also tested for ignitability, corrosivity and reactivity. The solidified material did not test positive for any of the above RCRA characteristics. However, Saltcrete that is not solidified would be considered ignitable and corrosive.

### Applicable EPA Hazardous Waste Numbers

The applicable listed EPA waste numbers for Saltcrete are as follows:

DELIVERABLE 2118, 2110, 2218, 2210
POMOCRETE SAMPLING & AMALYSIS PLAN
SALTCRETE SAMPLING & AMALYSIS PLAN

Hazardous Waste Number	Description
F001	Spent halogenated solvents used in degreasing
F002	Spent halogenated solvents
F003	Spent nonhalogenated solvents
F005	Spent nonhalogenated solvents
F006	Wastewater treatment sludges from electroplating operations
F007	Spent cyanide plating bath solutions from electroplating
F009	Spent stripping and cleaning bath solutions from electroplating operations where cyanides are used

### 1.2 Scope of Work

The following waste forms will be sampled in support of the Solar Pond/Pondcrete Stabilization Project:

- Pondcrete
- Saltcrete

The Pondcrete is segregated into two subgroups for sampling. The tri-walls are considered one group and the tri-walls in the metal containers will be the second group.

Saltcrete is divided into three subgroups. The tri-walls are one subgroup, tri-walls in metal containers are a subgroup, and 1/2 crates are a subgroup.

The purpose of the sampling effort is to obtain a sufficient number of samples to characterize each waste form. Specific goals of the waste characterization effort are as follows:

- To develop an analytical profile of each waste form such that, within a specified statistical confidence limit, each waste form can be characterized as a single population.
- To determine specific analytes that are known or suspected to be deleterious to cement chemistry reactions.
- To develop analytical values for specific analytes such that the capture efficiency of the final waste/cement formulations can be evaluated.
- To determine selected physical characteristics of the samples collected.

Additionally, samples of each waste form will be collected for treatability studies which will be conducted at the HALLIBURTON NUS laboratory in Pittsburgh, Pennsylvania. Details of the proposed treatability study are included in the Treatability Study Work Plan (HALLIBURTON NUS, 1991).

Table 1-1 provides a summary of the number of billets that will be sampled and the associated volumes that will be collected to accomplish the goals of the Sampling and Analysis Plan for waste characterization.

### 1.3 Sampling Strategy and Rationale

### 1.3.1 Introduction

In general, the goal of a sampling program is to collect a small but informative portion of the population being investigated. A representative sample is a sample that can be expected to adequately reflect the properties of interest of the entire media being sampled. As an integral part of the waste characterization and treatability studies, the objective of the sampling program of

REVISION 0 October 24, 1991

TABLE 1-1
VOLUME OF MEDIA TO BE COLLECTED DURING PONDCRETE AND SALTCRETE SAMPLING ROCKY FLATS FACILITY

	·	TABLE 1-1			<u> </u>	·	TABLE A	
WSTE SOURCE	LOCATION	TOTAL DISCRETE SAMPLE COLLECTED VOLUME PER BILLET	TOTAL DISCRETE VOLUME SKIPPED TO HALLIBURTON MUS FOR CHARACTERIZATION	TOTAL DISCRETE VOLUME TO BE COMPOSITED FOR TREATABILITY STUDY	TOTAL VOLUME OF COMPOSITE SAMPLE	VOLUME OF COMPOSITE SAMPLE TO BE STORED AT ROCKY FLATS	VOLUME OF COMPOSITE SAMPLE TO BE SHIPPED TO MALLIBURTON MUS FOR TREATABILITY STUDY	VOLUME OF COMPOSITE SAMPLE TO BE SHIPPED TO MALLIBURION MUS FOR CHARACTERIZATION
Pondcrete	Tri-valls - (16)	3.5 gal	2 gal	1.5 gal	St dal	12 gal	10 gai	2 gal
Ponderete	Tri-valls in Metals (40)	3 gal	2 gal	1 gal	24 gal	12 gal	10 gai	Z gal
Saltcrete	Tri-valls (42)	3.6 gal	2 gal	1.6 gal	64 gal	32 gal	30 gal	2 ga1
Saltcrete	Tri-valls in Metals (6)	13 gal	· 2 gel	11 gal	64 gal	32 gal	30 gal	2 gal .
Saltcrete	1/2 crates (12)	8 gat ;	2 gal	6 gal	64 gal	32 gal	30 gal	2 gol

this project is to obtain representative samples from each major waste source for specified field measurements and laboratory analyses. These samples will provide an evaluation of the physical and chemical properties of the waste, as they directly apply to the development of stabilization process formulations and the design of process equipment.

The waste forms considered for this sampling event include Pondcrete and Saltcrete. Because of the different nature of these waste forms and the availablility of previous information, samples will be taken using different approaches and will be analyzed separately. Therefore, sampling strategies need to be developed based on specific conditions to assure that the samples collected will give an accurate representation of each waste source.

To achieve the sampling objective, fundamental statistical concepts will be utilized where possible to develop sampling strategies to address the following issues:

- How many samples to take
- How to choose the sample
- How to estimate a population mean
- How to characterize the uncertainty in the estimate

4

### 1.3.2 Presurvey Estimate of Relative Standard Deviation

Formal sequential (multiphase) procedures are available which can guarantee, under certain conditions, achieving a prespecified boundary on the sampling error without previous knowledge of the population. Because only one sampling run will be conducted for this project, application of statistical formulas to determine the number of samples requires that previously obtained information on the population under consideration be available for evaluation. The previous sampling results will be utilized to provide rough

DELIVERABLE 2118, 2110, 2218, 2210 PONDORETE SUMPLING & ANALYSIS PLAN SALTORETE SUMPLING & ANALYSIS PLAN

estimates for relative standard deviations (RSDs) of a waste form. The RSD is estimated to be the ratio between the sample standard deviation and the sample mean, for each parameter to be analyzed in each waste form. Any analysis error incurred in making observations on sample units was negligible for the existing data.

The importance of a RSD, which is a measurement of the variability of a parameter in the population, for determining the sample size for each waste form is clear. The sample size required to characterize a population to a specified confidence level is related to the variability of the population. The sample size required to characterize populations which have relatively low variability would, in most cases, be expected to be smaller than those required to characterize a population in which the variability is higher for the same level of confidence.

### 1.3.2.1 Relative Standard Deviation of Existing Data

Pondcrete tri-walls and Pondcrete in metal containers were sampled for chemical and geotechnical data (Weston 1991). Fourteen samples of the Pondcrete tri-walls were collected and five Pondcrete samples from the metal containers were collected.

Summarized in Tables 1-2 and 1-3 are analytical results and general statistics for selected parameters for Pondcrete samples previously taken from tri-walls and metals, respectively. These parameters were selected based on their importance to the design of the development of stabilization process. Sample averages, standard deviations, relative standard deviations, and the 97.5% upper limit of each parameter's possible range were calculated.

. , '

TABLE 1-2 GENERAL STATISTICS AND ANALYTICAL RESULTS FOR TRI-WALLS ROCKY FLATS FACILITY

	_	<u> </u>	<sub>.</sub>		T	<del>-</del>	<del></del>	<del></del>	<u> </u>	_	T	7	==	<del>T</del>	<del></del>	<del>-</del>	Y	<del>-</del>		-
SOUTH	(1/80)	741000	2400000	395000	000015	000013	0000011	2,80000		0000336	4250000	0000797	4210000	0000997	0000697	000721	175.71	3/36/46.9	1982824.7	0.528
POTASSIUM	(1/60)	276000	3180000	236000	3200000	2730000	204000	2890000	104000	000000	0000272	264,0000	2720000	2530000	2570000	221000	2214412 9	1	1106916.5	0.499
BOROK		0	264.0	20	761	0.5	1350	20	o,	3	***	ς.	187	\$0	\$0	20	0 717		7.0.7	1.703
MERCURY (mg/kg)	(64/6m)	0.01	8.5	11.10	11.8	8.70	14.60	9.30	R	11 /0	25.	9.10	9.80	14.10	9.60	9.70	10.5	1	0.5	12.6
KICKEL (mg/kg)	78.2		0,,	675	897	677	\$15	403	679	017		75.6	663	\$4\$	252	217	7.927	174.2	3.07	263.7
1EAD (199/kg)	0 577		2.53	393.0	71.3	144.0	189.0	136.0	189.0	224.0		130.0	254.0	243.0	167.0	243.0	223.5	8	3,70	7 817
CHROMIUM (mg/kg)	2450	Cast	3	1720	1460	1370	1670	1300.	1990	886	07.01	2	1500	1580	824	1340	1488.7	413.2	0 278	2298.5
CADNIUM (mg/kg)	7630	1180		2050	2580	25.70	3380	2250	3400	1650	10,701		2810	2760	1420	2560	1.25.7	813.3	0 70B	4319.7
2-BUTANONE (vg/kg)	1900	1800	30.5	0061	2300	910	1700	077	710	1600	1800		1400	006	810	1700	1414.3	\$05.2	0.357	2.04.5
ACETONE (Ug/kg)	5700	\$ 100	00 / /		9009	0077	5800	3100	0062	7 800	\$800		2000	3900	3400	5200	4678.6	1034.9	0.221	0.707.0
SAMPLE	10-706	20 · 706	10-706		70-706	\$0. 204	90.706	404-07	750-01	750.05	750-03	Š	30.06	750.05	750.08	750 · 07	AVERAGE	5.0.	R.S.D.	97.5x «

DELIVERABLE 2118, 2110, 2218, 2210
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TABLE 1-2

GENERAL STATISTICS AND ANALYTICAL RESULTS FOR TRI-WALLS

ROCKY PLATS FACILITY

PAGE TWO

SAMPLE 10	CYAHIDE (mg/kg)	SULFATE (mg/l)	NITRATE (mg/l)	TOC (mg/kg)	MOISTURE (X)	PASS #200 (%)	NATURAL MOISTURE (X)	PENETROMETER (T/sq.ft)	TOTAL CALCIUM (mg/kg)	TOTAL CALCIUM (Ug/l)	SILVER (mg/kg)	ARSENIC (mg/kg)
904-01	96.3	1360.0	11000	15100	63.9	76.5	181.4	0.00	337000	2500	147.0	7.60
904-02	93.1	1770.0	11700	21900	59.6	86.0	180.4	0.00	202000	7350	92.3	6.00
904 - 03	123.0	1190.0	9820	52600	69.8	93.0	229.1	0.00	235000	2500	103.0	11.10
904 - 04	49.7	1160.0	10700	21100	63.5	77.4	181.1	0.00	167000	<i>7</i> 780	96.0	2.75
904 - 05	43.8	952.0	12300	23600	61.5	89.3	162.6	1.70	337000	28500	81.2	12.50
904 - 06	107.0	1060.0	5040	11100	61.5	60.9	159.0	0.00	139000	6270	102.0	4.90
904 - 07	43,4	1440.0	13100	16900	59.5	93.2	161.3	1.70	247000	23200	77.1	11.20
750-01	48.4	1190.0	11000	8640	61.1	86.4	158.1	1,30	328000	21400	119.0	2.55
750-02	36.8	1310.0	8080	17800	64.4	80.0	204.0	0.00	125000	12500	64.2	6.80
750-03	58.2	1260.0	9380	15100	64.1	86.7	196.0	0.75	189000	21900	55.9	5.90
750-04	29.4	1330.0	9330	13700	54.9	54.1	140.9	0.00	188000	12500	2.2	4.70
750-05	50.4	363.0	10400	21300	60.5	87.2	153.7	1.50	335000	44000	93.1	5.10
750-06	52.4	822.0	11100	18500	61.5	73.6	163.5	3.00	142000	27400	59.8	2.15
750-07	40.1	1320.0	7210	18900	61.8.	81.9	165.2	0.00	180000	2500	79.2	6.50
AVERAGE	62.3	1180.5	10011.4	19731.4	62.0	80.4	174.0	0.71	224857.1	15735.7	83.7	6.4
\$.D.	29.5	325.4	2122.9	10359.3	3.3	11.4	23.3	0.97	78739.0	12382.9	33.6	3.3
R.S.D.	0.474	0.276	0.212	0.525	0.054	0.142	0.134	1.364	0.350	0.787	0.402	0.507
97.5x <	120.1	1818.2	14172.3	40035.7	68.5	102.9	219.6	2.6	379185.5	40006.1	149.6	12.8

Source: Weston 1991

Data reported in mg/kg and  $\mu$ g/kg are concentrations in the solid waste. Data reported in mg/l and  $\mu$ g/l are concentrations in the waste leachate.

TABLE 1-3 GENERAL STATISTICS AND ANALYTICAL RESULTS FOR METALS ROCKY FLATS FACILITY

SWPLE	ACE TOKE (199/kg)	2-8UTANONE (19/kg)	CADHIUM (mg/kg)	CHRONIUM (mg/kg)	(#4/kg)	WICKEL (mg/kg)	MERCURY (mo/kg)	BORON	POTASSIUM	SOULH	CYANIDE
750.08	9029	2200	2420	0911	20.8.0	181	3	1	(1/20)	(1/8)	(mg/kg)
750.09	2900	078	RSR	101			8.	2	2330000	3590000	33.9
750 · 10	2900	97		arc .	2/1.0	136	6.70	0,	2300000	3930000	38/5
		200	0012	1050	223.0	432	8.40	1880	2760000	0000257	77 1
750 - 11	069	65	3850	1970	0 177	777	11				
750.13	į					3	13.70	175	2390000	2650000	38.6
31 -07 -	0//	55	1850	1040	303.0	338	11.60	919	2690000	0000077	
AVERAGE	2692.0	244.0	2215.6	1110 2	7 161	0 701					23.5
					. 563.0.	8.00°	7.01	8.029	2494000.0	3798000.0	1.33
5.0.	2241.4	280.2	1084.3	561.9	74.6	0 181					
0	118				,	0.00.	ζ.,	1.867	214779.0	729362.7	18.6
	0.033	1.183	0.489	0.502	0.236	0.475	0.274	1,191	0.086	0 102	(23)
97.5x c	7085.1	1.6972	4340.9	2220.5	473.3	0.72	1 41	30406	2,707.00		235.
						?	- 1	C.VOV. 2	6714700.8	5227551.0	80.5

<u>.                                      </u>												
	SAMPLE 10	SULFATE (mg/l)	WITRATE (mg/l·)	10C (mg/kg)	HOISTURE (X)	PASS #200	MOISTURE (X)	PENETRONETER (17/1/2)	TOTAL CALCIUM	CALCIUM	SILVER	ARSENIC
	750.08	1160.0	8010	22000	56.1	8.8	131.3	1 25	182,000	(1/8)	(mg/kg)	(mg/kg)
	750.09	79.9	7760	7680	62.2	9.09	148.8	05.7	00000	00000	73.6	8.5
	750 · 10	1270.0	11400	16600	58.3	79.3	135.7	36.1	00001	0000	29.5	18.1
	750-11	1210.0	۲۶۵۵	23800	56.4	61.3	5 19	03.6	00000	0167	102.0	1.85
1	750-12	1360.0	10000	23100	57.9	77.77	137.7	3, 1	000031	מסבני	124.0	80 80
	AVERAGE	1016.0	8334.0	18636.0	58.2	7.17	123.2	,	000461	00000	9.6	8.~
Ц_								3.3	0.00001	3,464.2.0	۸. 8	0.6
	S.D.	528.6	2612.9	6747.0	2.4	9.8	34.5	1.4	41445.1	6 76705	4 A	9
	R.S.D.	0.520	0.314	0.362	0.042	0.137	0.280	189.0	0 244	126.1		0.5
	97.5x <	2052.0	13455.4	31860.2	63.0	91.0	190.9	6.4	250032.5	138611.9	149.8	20.7
ية لا	urce: Veston	1661										

Source: Veston, 1991 Data reported in mg/kg and kg/kg are concnetrations in the soild waste. Data reported in mg/l and μg/l are concentrations in the waste leachate.

DELIVERABLE 2118, 2110, 2218, 2210
PONDORETE SUMPLING & AVALYSIS PLAN
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As shown in the tables, some parameters have very high RSDs; however, the final result of the stabilization process may not be sensitive to those parameters when compared to other considerations. Instead of using the highest RSD to set the sample size, it was determined that a more practical approach is to use the parameters that will be most likely to cause failure of the TCLP tests after the stabilization process. A conservative estimate of constituent concentration in the waste extract (CCWE) was made based on a 60% dilution (based on weight) of Pondcrete during stabilization (assumes a baseline 3:1:1 mixing ratio between Pondcrete material, cement, and water in the stabilization process), a 20:1 dilution during the standard TCLP test, and assuming 100% of the contaminant will leach. equation was used to estimate the leachate concentrations of each contaminant after the stabilization process:

$$CCWE(mg/1) - C_s(mg/kg) \times \frac{3}{5} \times \frac{1}{20}$$

Where: CCWE (Constituent Concentrations in Waste Extract) is the leachate concentration and C<sub>s</sub> is the 97.5% upper limit of possible original concentration.

The estimated CCWEs were then compared with the regulatory level for each regulated contaminant, respectively. Table 1-4 summarizes these results. In both tri-walls and metals, cadmium has the highest CCWE/standard ratio and will be used to determine the data quality objectives for sampling.

### 1.3.3 Data Quality Objectives for the Sampling

Data Quality Objectives (DQOs) are statements that provide the critical definitions of confidence required in drawing conclusions from the entire project data. These objectives determine the degree of total variability (uncertainty or error) that can be tolerated in the data. As both sampling and analysis error contribute to the overall uncertainty of data, these limits of

# TABLE 1-4 COMPARISON OF ESTIMATED CCMEs WITH REGULATORY LEVEL PONDCRETE ROCKY FLATS FACILITY

TRI-WALL

80.0	\$2.58	[8.5]	69.69	£9°72	95.21	05.2961	01.0	75.0	OITAR
0.2	570.0	5.0	52.0	15.0	5.2	990.0	0.027	0.068	ORAGKATZ
7.0	5.3	\$.0	2.55	9.51	0.98	9.951	1.57	5,105	EST, CCVE
8.51	9.671	2.71	2.827	7.812	2.8955	7.9183	5. 2025	0.7073	> X5, 19
102.0	207.0	175.0	985.0	977.0	875.0	895.0	722.0	0,221	.0.2.8
(,(	3.22	3.8	5.921	9.65	2.812	2.218	5.202	6.2501	.0.2
7.9	7.28	2.01	7.917	2.855	7.8811	1.2575	ולול' ב	9.8732	AVERACE
05.8	5,97	07.9	217	0.255	· 07£1	0952	0021	002\$	10-027
21.5	8.92	09.9	555	0.731	728	1620	018	3,000	90-051
01.2	1.29	01.21	575	0.215	0851	0975	006	1000	\$0.0\$7
04.3	5.5	08.9	567	0.125	0051	2810	0071	000\$	70.051
06.8	9.22	01.9	778	0.821	0701	0761	0081	0085	20.027
08.8	5. 29	07'11	310	0.255	886	0591	0091	0087	50-027
22.5	611	٣.0	679	0.981 .	0661	2,000	012	5000	10.052
05.11	1.77	9.30	507	0.351	1200	5550	011	2100	20-706
06.3	201	09.21	SIS	0.981	0291	3380	0071	2800	90 - 706
02.51	5.18	07.8	677	0. 221	0251	0725	016	0077	\$0.509
27.5	96	06.11	897	2.17	0971	2580	5200	0009	70-706
01.11	103	01.11	675	193.0	0271	2020	0051	0077	₹0 - 706
00.8	2.59	09.01	867	0.815	0851	3160	0081	0015	20-706
09.7	171	00.81	587	0.22	0572	0197	0061	0018	10-706
(4/50)	(w3/k3) ·	(64/64)	(mg/kg)	(6x/6u)	(6x/6us)	(6x/5u)	(nð\kg)	(63/60)	01
ARSENIC	SILVER	KERCURY	HICKEL	QX3J	CHROHIUM	CADKIUK	2-BUTANONE	3HO133A	3 JAHA2

Estimated CCVE assumes 100 percent leaching of constituent, 60 percent dilution during stabilisation, and 20:1 dilution in TCLP test.

TABLE 1-4 COMPARISON OF ESTIMATED CCWES WITH REGULATORY LEVEL PONDCRETE ROCKY FLATS FACILITY PAGE TWO

	F	_	Ŧ	_	_	_	<del>-</del> -	_	_	_	_	_	_	_	7	_	_	_	_	_
	ARSENIC	(04/00)		8.8	18.1		1.85	80.00	8.2		0.6		۶.8	779 0	95	\$.03	9 0		^	
	SILVER	(mq/kg)	F	7.6	29.5	501	301	151	9.69		۲. ۶	0 52	23.8	677.0	8 0/1	3	5.7	5	0.076	62.43
	MERCURY	(mg/kg)	9	3	6.70	07 8		13.8	11.60		10.4	2 0	;	0.274	1 91		0.5	02.0	23.0	2.41
	MICKEL	(mg/kg)	381		136	432		Š	338		286.8	183.8	?!	0.475	747.0		22.6	0.32	100	70.03
	15.00	(mg/kg)	298.0		0.175	223.0	0 267	0.00	503.0	1 261	363.0	7.92		0.736	473.3		7.31	0.51	37 B/	5.5
	CHROMIUM	(61/61)	3	701	2	1030	10701		1040	1110 2	,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	561.9	63 6	0.300	2520.5	7 77	8.0	5.5	12.81	
		/83/2	0277	828		2007	3850	0381	000	2215.6	, , ,	1004.3	087 0		4.340.9	130.2	3:20	0.088	1973.14	
2. Distances	(vo/ko)	2200	0022	0%8	3	200	9	\$\$		0.22	2 A8A 2	3.000	1,183	. 0,,,,	(40).1	1.17	*	067	0.10	
ACEJORE	(100/10)	9700		2900	2000		069	770		2692.0	7 172		0.833	7085 1		212.6	8	2	0.36	
SAMPLE	01	750.08	20 02	750.09	750 · 10	11.0%	11.00	750-12	23.63.77	AVEXAGE	5.0.		R.S.O.	97.51		EST. CCVE	STANDARD		KAT 10	

DELIVERABLE 2118, 2110, 2218, 2210
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variability must be incorporated into the sampling and analysis plan and achieved with detailed sampling and analysis protocols.

The standard error estimates and confidence intervals presented for the sampling strategy will reflect only uncertainty due to sampling error, that is, the error associated with the fact that only a sample, rather than the whole population, is observed. This assumes that the sample is representative of the entire waste form.

By defining the sampling DQOs separately from the overall project DQOs, the sampling protocols can be developed using simple statistical concepts to achieve the specified quantitative DQOs for sampling in each waste standards for sampling errors. form will be defined as relative percent error, i.e., the magnitude of tolerable sampling error is expressed in relative terms as a percent of the quantity to be estimated. An initial value of the sampling DQO is selected as 15% error of the sample mean. percent error was selected because the number of samples required to achieve this DQO is reasonable based on schedule and cost. Additionally, a greater number of samples does not decrease the DQO error in a significant manner until a very large sample population is selected (i.e., large increases in sample size results in small decreases in DQO error). Further discussion is provided in Section .........

### 1.3.4 Determination of Sample Size

As mentioned earlier, statistical approaches will be used to determine the sample size required to generate data which satisfy the specified sampling DQO.

For random sampling of a finite population, the formula for standard error of the estimator of population mean specifies a relationship between sample size n and the uncertainty of the estimation (Wadsworth, 1990). This relationship can be used to

DELIVERABLE 211B, 211C, 221B, 221C POMOCRETE SAMPLING & ANALYSIS PLAN SALTCRETE SAMPLING & ANALYSIS PLAN

determine the sample size required to obtain an estimate with a desired level of precision. Given a DQO, expressed in a relative percent error, and the estimated RSD of the population, under 95 percent confidence limit the sample size can be determined as

$$n = n_0 / [1 + (n_0/N)]$$

Where:

$$n_o - \left(\frac{z \cdot RSD}{DQO}\right)^2$$

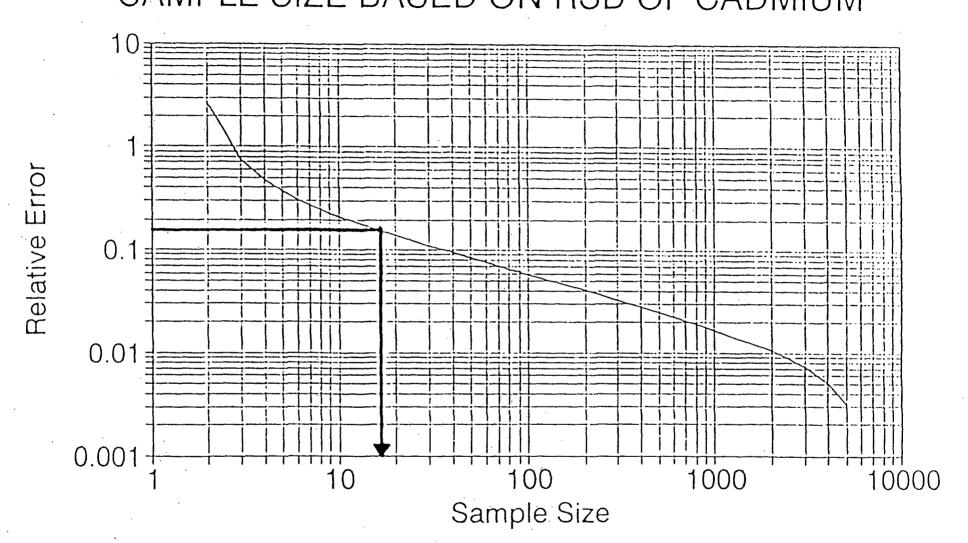
N is the population size (total number of billets in a waste form), and z is the 2.5 percent quantile from Student's t Distribution with n-1 degree of freedom.

This equation results in collecting more samples than what would be required to be collected using the equations recommended in SW-846; therefore, this methodology will exceed the minimum sampling requirements of SW-846. This approach was taken to satisfy the technical needs and better quantify the uncertainties of the project.

### 1.3.4.1 Pondcrete Sample Bize

As mentioned previously, cadmium was determined to be the parameter which may cause the most concern for the TCLP analysis of the solidified product. Therefore, the RSDs calculated for cadmium were used to determine the required sample sizes for Pondcrete triwalls and for Pondcrete in metal containers. Figures 1-1 and 1-2 were developed, based on formulas described earlier, to assist the selection of sample sizes based on RSDs of cadmium and the total numbers of billets for tri-walls and metals, respectively.

# TRIWALL (TOTAL OF 5806 BOXES). SAMPLE SIZE BASED ON RSD OF CADMIUM

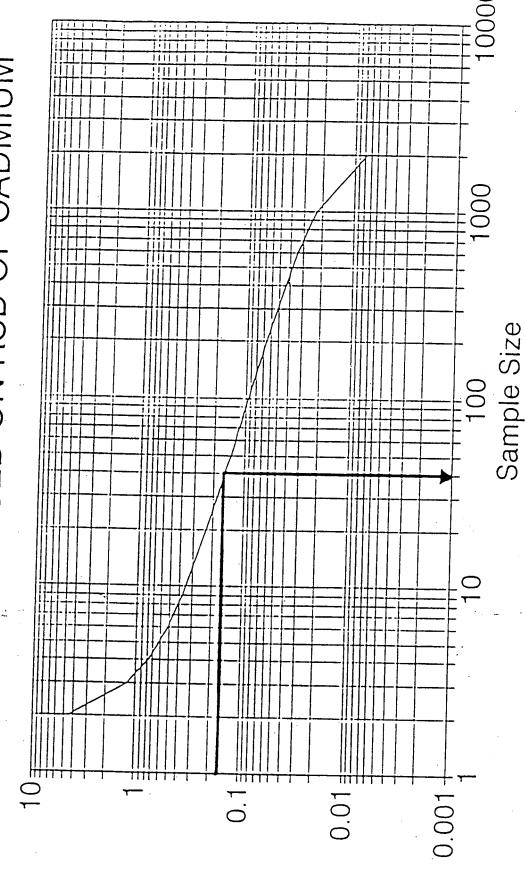


- RSD = 0.298

Figure 1-1 Comparison of Relative Error to Sample Size (Triwalls)

# METAL (T SAMPLE SIZ

(TOTAL OF 2293 BOXES) SIZE BASED ON RSD OF CADMIUM



Comparison of Relative Error to Sample Size (Metals) Figure 1-2

> 0.489 RSD

Relative Error

DELIVERABLE 2118, 2110, 2218, 2210
PONDORETE SAMPLING & AVALYSIS PLAN
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As shown in the Figures, using a relative error of 15%, the required number of samples for tri-walls and metals are 16 and 40, respectively. As can be seen in Figures 1-1 and 1-2, relatively large changes in sample sizes are required to decrease the DQO error.

### 1.3.4.2 Saltcrete Sample Size

Statistical concepts based on previous analytical data are not applicable to the Saltcrete billets because existing data for critical parameters (metals, anions) do not exist. Because of the lack of data for Saltcrete, uncertainties exist for the chemical composition of Saltcrete. Therefore, it will be assumed the Saltcrete is more variable than Pondcrete and will require more samples per total population size to accommodate potentially larger variances.

Because the schedule is relatively short for this project, only one sampling round will be conducted for Saltcrete. Therefore, sample size must be as large as possible to ensure that quality data is obtained to adequately characterize the Saltcrete. Sixty samples is considered to be the maximum sample size that can be accommodated because of the short schedule. Sixty samples is believed to be sufficiently large to account for variances in the Saltcrete. Essentially, 60 samples for Saltcrete, compared to 56 samples for Pondcrete, represents almost three times as many samples when compared to a total population of 2,936 and 8,099, respectively.

The 60 samples for the total population of Saltcrete will be subdivided into three groups. The three subgroups will be triwalls, 1/2 crates, and tri-walls in metal containers. The number of samples for each subgroup were determined by proportioning the total number of samples between the three subgroups based on the total number of billets (i.e., tri-walls, 1/2 crates, or tri-walls

DELIVERABLE 2118, 2110, 2218, 2210 PONDORETE SAMPLING & ANALYSIS PLAN SALTORETE SAMPLING & ANALYSIS PLAN

in metal containers). The number of samples per subgroup were also proportionally divided between the 904 Pad and the 750 Pad. This method provided the following number of samples for each subgroup:

	750 Pad	904 Pad
Tri-walls	14	28
1/2 Crates	12	0
Metal	2	4

### 1.3.5 Uses of Statistical Computer Package

SYSTAT/SYGRAPH 5.0 with DESIGN and SAMPLE modules, a comprehensive statistics, graphics, and data management package for IBM-PC compatibles, will be utilized throughout the entire sampling task. This computer package can perform tasks ranging from simple statistical calculations to design of highly complex sampling that use stratification, clustering, and variable probabilities. The use of a statistic computer package saves time, reduces possible human errors and produce high quality graphic outputs.

### 1.3.6 Sample Design for Pondcrete and Saltcrete

Because of the way the waste containers are stored (i.e., large stacks in separated tents), it is desired to have a representative sample consisting of containers from every storage area and layer of stacks without moving too many containers. Given the relatively small size of sample (i.e., approximately 60 each for Pondcrete and Saltcrete), a simple random sampling approach clearly cannot assure that samples are selected from the middle of a stack or from only some of the tents. Therefore, the predetermined numbers of waste samples required for the treatability study were selected by a systematic sampling approach. This approach was designed to obtain samples from every portion of the waste storage areas/layers but also consider the accessibility of selected containers.

The number of samples required to achieve the DQO was initially developed by assuming a simple random sampling approach would be used. The following assumptions were considered thereby allowing the same number of samples to be used with a more controlled sampling approach:

- There is no statistically significant spatial patterns of the waste characteristic in a layer of any stack (i.e., the underlying probability distributions of the characteristics is stationary within a layer).
- The waste characteristics are independent of the different layers in a stack.
- The waste characteristics are independent of stacks in a tent and between tents or laydown areas.

The first assumption allows containers to be selected from corners or outsides of a stack so it is not necessary to move many container in order to take a container in the middle of a stack. With the second assumption, containers of all layers at a selected location in a stack can be collected simultaneously. The third assumption supports using tents and stacks as two levels of sampling clusters in the multi-stage cluster sampling approach.

### 1.3.6.1 Pondcrete Sampling

The multi-stage cluster sampling approach was accomplished in the following steps for Pondcrete:

• Develop maps showing the layout of the two waste storage pads, locations of stacks in the tents and outside laydown areas, and detailed drawings of positions of every container in a stack. All the maps and drawings are included in Appendix A and B.

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- Identify the waste type and form for every container on the drawings.
- Verify the numbers of containers against the list of inventory.
- Select and mark potential containers or groups of containers to be sampled from each stack considering the size of the stack and the accessibility of the containers. These groups are located at corners or outsides of a stack and consist of overlaying containers from each layer of the stack.
- A random number table was used to generate random choices whenever a cluster (tent or stack) or waste container selection was performed in the following steps.
- The Pondcrete Tri-wall samples were selected from tents that contain mostly this type of waste (i.e., tents 9, 10, 11 in 904 Pad and tents 3, 4, 5 in 750 Pad). One stack was selected from a tent first, then one group of containers among the previously determined potential sampling groups of this stack was chosen. This procedure was repeated for each tent listed above. Overall, 16 Pondcrete Tri-walls were selected.
- The Pondcrete metal containers were located in one outside area and three stacks in Tent 9 on the 904 Pad and 27 double-layer rows in the south and north laydown areas on the 750 Pad. Each metal container usually has three Pondcrete Tri-walls inside. The sampling was performed by selecting stacks or rows then containers similar to the Triwall sampling. Overall, 14 metal containers with 42 Pondcrete samples were selected.

### 1.3.6.2 Saltcrete Sampling

The multi-stage cluster sampling approach was accomplished in the following steps for Saltcrete:

No previous information was available to determine specific numbers of samples required for each container type to achieve a given DQO. Therefore, the total Saltcrete sample number (i.e., 60) was divided among waste storage areas and the three different waste-types by using simple proportions. The following table shows the numbers of samples to be collected from each waste-form on each pad.

Waste-forms	750 Pad	904 Pad
Tri-wall	14/771	28/1544
Half-Crate	12/675	0/0
Metal	2/102	4/210

NOTE: Sample number/total Saltcrete number

- The Saltcrete Tri-wall samples were selected from tent 8 in 904 Pad and tents 2 and 6 in 750 Pad. Similarly, stacks were selected first then followed by sample groups. Overall, 42 Tri-wall samples were selected.
- The Saltcrete half-crates were stored in the south laydown area in 750 Pad. Following the stack then sample group procedure, 12 half-crates were selected.
- The Saltcrete metal containers usually have two Saltcrete billets in one container. These containers were located in two outside areas and one stack in tent 8 in 904 Pad and two stacks in the south laydown area in 750 Pad. Overall, 3

metal containers with 6 Saltcrete samples were selected following the stack then container procedure.

 The times that these selected Saltcrete samples were produced was identified and a histogram (Figure 1-3) developed to determine the variation of the production over time.

Figure 1-3 was produced after EG&G personnel located the Saltcrete billets that were selected for sampling and determined their dates of production. Of the 42 Tri-walls selected, 18 had production dates that were accessible to EG&G personnel. These Tri-walls are evenly distributed with time over the period in which Tri-walls were produced. The remaining unknown Tri-walls shown in Figure 1-3 may be characterized with time during sampling or possibly after analytical results are obtained.

Of the 24 unknown Tri-walls, 15 Tri-walls may have a production date on a side of the Tri-wall that cannot be observed because it is adjacent to another Tri-wall. During Tri-wall mobilization the Tri-walls will be examined to see if any production dates are visible.

The remaining 9 unknown Tri-walls have serial numbers but no production date on the outer packaging. If possible, these Tri-wall's production dates could be determined from historical production log books. If this is not possible, then upon receiving the analytical data from characterization, this information will be compared to the data from the Tri-walls with known production dates to determine if any correlations exist.

The half-crates that have known production dates are evenly distributed with time. Two half-crates do not have production dates which is not considered to be a significant concern.

DELIVERABLE 211B, 211C, 221B, 221C POMDCRETE SAMPLING & ANALYSIS PLAN SALTCRETE SAMPLING & ANALYSIS PLAN

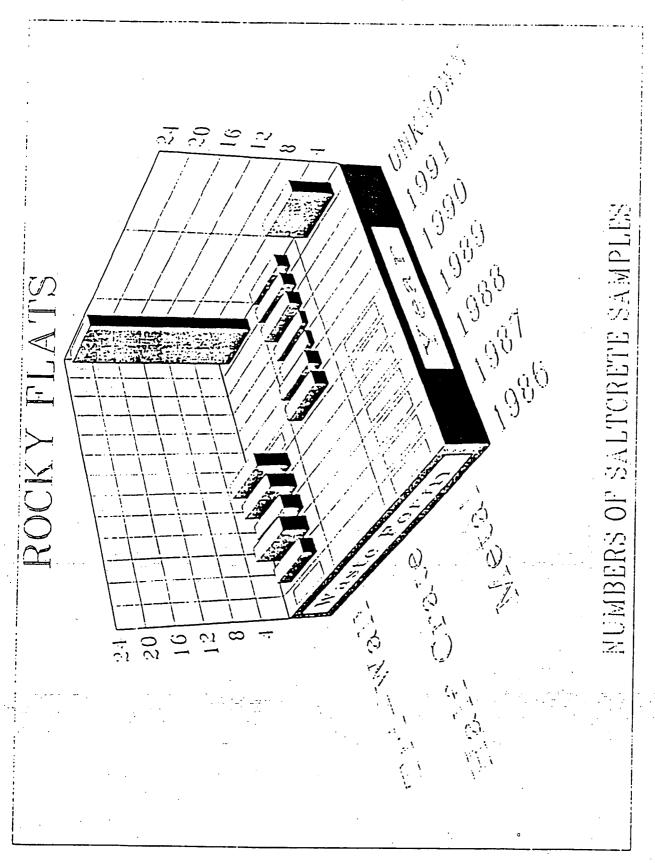


FIGURE 1-3 HISTOGRAM OF WASTE FORMS
FROM
YEARS 1986 TO 1991

DELIVERABLE 2118, 211C, 2218, 221C POMOCRETE SUMPLING & ANALYSIS PLAN SALTCRETE SAMPLING & ANALYSIS PLAN

None of the three metal containers selected for sampling have known production dates. However, because the billets in metal containers are a subset of the Tri-walls, this may not be a major concern. When the metal containers are opened during sampling, production dates may be distinguishable on the Tri-wall packing. When analytical data is obtained for the Tri-walls in the metal containers, it will be compared to the data for the Tri-walls.

Appendices A and B illustrate the locations of all the selected samples. When the selected samples are against the wall and not easily accessed, optional (alternative) sampling locations are identified.

### 1.4 Sample Analyses and Rationale

The purpose for conducting this sampling and analysis program is to provide input to the solidification formula development and is required to address various regulatory concerns, mainly the Land Disposal Restrictions (LDRs) from 40 CFR 268 and 49 CFR packaging and shipping requirements. A brief overview of the LDRs is provided below.

### 1.4.1 Land Disposal Restrictions

The land disposal restriction (LDR) requirements (40 CFR 268) apply to all hazardous wastes as designated by the U.S. EPA. The LDR regulations specify treatment standards that must be met prior to land disposal of hazardous waste. Treatment standards are expressed as a concentration limit in an extract of the waste, as a concentration limit in the waste, or as a specified technology. If a technology is not specified, any method of treatment may be used as long as the treatment standard is met.

The EPA Hazardous Waste Numbers associated with the Pondcrete and Saltcrete waste are F001, F002, F003, F005, F006, F007, and F009.

DELIYERABLE 2118, 211C, 2218, 221C PONDORETE SAMPLING & ANALYSIS PLAN SALTCRETE SAMPLING & ANALYSIS PLAN

EPA Waste Code D006 is also appropriate for Pondcrete. The LDR treatment standards for these waste codes are provided in Table 1-Most treatment standards are expressed as concentration in thewaste extract (mg/l), and some are expressed as concentration in If two treatment standards are given for a particular constituent (i.e., cadmium), the lowest value applies. noted that for F005 waste, the treatment standard 2-nitropropane and 2-ethoxyethanol is incineration. That is, if the solvent used was 2-nitropropane or 2-ethoxyethanol, then the waste must be incinerated or a variance from the treatment standard could be sought. However, these chemicals are not anticipated to be present based on process knowledge. If they are found to be present, EG&G shall be notified immediately.

The treatment standards for mixed (hazardous/radioactive) waste are whatever is specified for the corresponding nonradioactive hazardous waste. There are a few exceptions to this; however, they do not apply to the waste sources associated with this project.

In summary, the treated Saltcrete and Pondcrete, after solidification, must meet the treatment standards in Table 1-5 prior to land disposal. Therefore, the raw (untreated) waste should be analyzed for the constituents presented in Table 1-5. If any of these constituents are not present in the untreated waste, or are present below the LDR treatment standard concentration limits, there is no need to further analyze the solidified (treated) waste for such constituents.

### 1.4.2 Specific Analyses and Rationale

Analysis for the LDR-regulated organics will be conducted on each waste source. This analysis is being conducted to determine if the total amount of each compound in the waste sources is sufficiently low such that, when considering the TCLP procedure, the maximum possible leachate concentration is below the regulatory values in

DELIVERABLE 211B, 211C, 221B, 221C POMDCRETE SAMPLING & ANALYSIS PLAN SALTCRETE SAMPLING & ANALYSIS PLAN

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### LDR TREATHENT STANDARDS PONDCRETE AND SALTCRETE ROCKY FLATS FACILITY

ROCKY FLATS FACILITY  LDR Treatment Standard (Nonwastewaters)				
Design	LUK 11	eatment Standard	T	l ers)
Regulated Hazardous Constituent	F001-F003	F005	F006, F007, F009	D006
Acetone	0.59 mg/1 <sup>(1)</sup>	0.59 mg/l	АИ	NA
n-butyl alcohol	5.0 mg/l	5.0 mg/l	Ай	AN
Carbon disulfide	4.81 mg/l	4.81 mg/l	AN	АИ
Carbon tetrachloride	0.96 mg/l	0.96 mg/l	NA	АИ
Chlorobenzene	0.05 mg/l	0.05 mg/l	AN	АИ
Cresols and Cresylic Acid	0.75 mg/l	0.75 mg/l	AN	АИ
Cyclohexanone	0.75 mg/l	0.75 mg/l	АИ	АИ
1,2-dichlorobenzene	0.125 mg/l	0.125 mg/l	АИ	АИ
Ethyl acetate	0.75 mg/l	0.75 mg/l	NA	АИ
Ethyl benzene	0.053 mg/l	0.053 mg/l	AN	АИ
Ethyl ether	0.75 mg/l	0.75 mg/l	AN	NA
Isobutanol	5.0 mg/l	5.0 mg/l	AN	NA
Hethanol	0.75 mg/l	0.75 mg/l	NA	АИ
Methylene chloride	0.96 mg/l	0.96 mg/l	NA .	NA
Methyl ethyl ketone	· 0.75 mg/l	0.75 mg/l	NA	AN
Methyl isobutyl ketone	0.33 mg/l	0.33 mg/l	NA NA	АИ
Nitrobenzene	0.125 mg/l	0.125 mg/l	NA	NA
Pyridine	0.33 mg/l	0.33 mg/l	NA	NА
Tetrachloroethene	0.05 mg/l	0.05 mg/l	NA	AN
Toluene	0.33 mg/l	0.33 mg/l	NA	NA
1,1,1-trichloroethane	0.41 mg/l	0.41 mg/l	NA	NA
1,1,2-trichloro-1,2,2- trifluoroethanu	0.96 mg/l	0.96 mg/l	АИ	АИ
Trichloroethene	0.091 mg/l	0.091 mg/l	NA	NA
Trichlorotrifluoromethane	0.96 mg/l	0.96 mg/l	NA	NA
Xylene	0.15 mg/l	0.15 mg/l	АИ	. NA
1,1,2-trichloroethane	7.6 mg/kg <sup>121</sup>	7.6 mg/kg	АИ	NA ·
Benzene	3.7 mg/kg	3.7 mg/kg	АИ	NA
2-nitropropane	AN	Incineration <sup>(3)</sup>	AN	АИ
2-ethoxyethanol	NA	Incineration	АИ	АМ
Cyanides (total)	NA	АИ	590 mg/kg	NA
Cyanides (amenable)	NA	NA	30 mg/kg	AN
Cadmium	NA	AN	0.066 mg/l	1.0 mg/l
Chromium (total)	NA	. NA	5.2 mg/l	. NA
Lead	NA	NA	0.51 mg/l	АИ
Nickel	NA	NA	0.32 mg/l	NA .
Silver	АИ	NA	0.072 mg/l	АИ

mg/l - concentration in waste extract
 mg/kg - concentration in waste
 Specified treatment technology

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40 CFR 261, Subpart C for toxicity characteristic and values in 40 CFR 268, Subpart D, Land Disposal Restrictions. Therefore, depending on the analysis results, the analysis of the stabilized waste for certain compounds may not be required prior to final disposal.

Metals analysis will be conducted for both waste forms and will include those metals regulated by 40 CFR 261.24 (toxicity characteristic), plus nickel and boron. Total metal content and TCLP analysis will be performed for each parameter. The toxicity characteristic metals will be analyzed for regulatory purposes; nickel will be analyzed because it is a constituent of F006-type wastes (electroplating wastes), which is applicable to the waste forms, and boron will be analyzed because it can interfere with cement chemistry. Cyanide will be analyzed because it is a constituent of F006-type wastes.

Both Pondcrete and Saltcrete will be analyzed for ammonia and total organic carbon. Both of these parameters, depending on their concentrations, can affect cement chemistry.

The wastes will be analyzed for alkalinity, potassium, calcium, magnesium, and sodium. These parameters will provide input to developing the waste/cement formulation.

An ASTM (D3987-85) leach test will be conducted on the Saltcrete and Pondcrete. The leachate will be analyzed for phosphate, sulfate, nitrate, chloride, and total dissolved solids. This analysis will determine the amount, if any, of these compounds that will redissolve. Additionally, these compounds can affect the cement chemistry.

Gross alpha and gross beta will be analyzed on each waste form to characterize the activity level of the waste.

DELIVERABLE 211B, 211C, 221B, 221C PONDORETE SUMPLING & AVALYSIS PLAN SALTORETE SUMPLING & AVALYSIS PLAN

Several geotechnical parameters will be analyzed to characterize the physical condition of the solid waste. Percent moisture, bulk density, and specific gravity are common physical parameters for characterization of the waste source. The Blaine fineness test provides an indication of the fineness of the material based on the permeability of air. The Atterberg limits will provide an indication of the plasticity of the material. Particle size analysis will determine the distribution of the material size and the swell test will determine if dry material will expand when exposed to water. Disaggregation testing will determine if the material will dissolve when exposed to water.

Unconfined compressive strength will provide an estimate of the waste's current strength. Also, comparisons with other chemical parameters may be possible to develop correlations that will indicate if a particular parameter affects strength. Cement content will provide a rough estimate of the ratio of cement to waste. Petrographic analysis will provide qualitative analysis of the current structure of Pondcrete and Saltcrete. Information pertaining to mixing, unhydrated cement, and cement formation can be provided by petrographic analysis.

100 mg 100 mg